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VIBRATORY GRINDING AND POLISHING OF METALLOGRAPHIC SPECIMENS

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VIBRATORY GRINDING AND POLISHING OF METALLOGRAPHIC SPECIMENS

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S. Matras

Metallurgy Division

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ABSTRACT

The range of metallographic specimen preparation by vibratory polishing has been extended to include the grinding operation. A controlled method has been developed that reduces the metallographic grinding operation to essentially one step. This was achieved by: (1) controlling the horizontal and vertical components of vibration by changing the springs and the weight of the bowl of the apparatus to conform to the specific requirements of grinding, and (2) providing a special fixture that guides the specimen in a controlled and predetermined manner.

The grinding fixture consists of a Lucite bowl that holds an 8-in.-diam, silicon carbide paper disc and a rotating turret in which the specimens are spaced and loaded with appropriate weights. The grinding produces specimens whose surfaces possess a minimum of disturbed metal with virtually no embedded abrasive. The specimens have flat surfaces and their edges are well-preserved. Inclusions and second phases, not normally in evidence at this stage of specimen preparation, are clearly delineated in the matrix. Retention of hard inclusions in a soft matrix is excellent.

The commercial vibratory polishers do not always produce a scratch-free final polish, especially with high-purity materials that are soft and ductile. To achieve satisfactory surfaces, a final-polishing fixture utilizing a center-mounted, rotating carrier was devised. This fixture replaces the standard, stainless-steel, polishing bowl and retaining ring. The fixture is mounted on a standard vibratory polishing unit. In contrast to standard vibratory polishing units that require specimen loadings of 300 to 600 g, the new device may be operated without any load on the specimen and will produce a superior polish.

Preparing metallographic specimens now involves only three steps: specimen grinding on 400- or 600-grit silicon carbide paper, intermediate polishing, and final polishing. No more hand operations are necessary. The method has broad applicability. For example, it is routinely used for the specimen preparation of cermets, and high-purity thorium and thorium-plutonium alloys have also been successfully prepared. The technique is presently adapted to glovebox work for plutonium and its alloys, diffusion couples and alloys, and cermets that are difficult to prepare.

INTRODUCTION

The introduction of the vibratory metallographic-specimen preparation by Krill[1] in 1956 was a significant departure from the conventional metallographic technique. The method is a form of automation. Most previous approaches to the automating of the conventional method imitated the manual procedure and therefore did not change the basic difficulties inherent in manual preparation. Indeed many metallographers still prefer manual grinding and polishing of critical and difficult materials to avoid surface deformation and the creation of defects caused by mechanical methods. With the need for examination of radioactive materials, the development of automatic methods became imperative and vibratory specimen preparation looked highly attractive. The vibratory process was developed by Long and Gray[2-4] with the help of Meador[5] and was further developed by Rothstein and Turner[6] who presented a detailed analysis of the principles of vibratory polishing. The process was adopted by many U. S. laboratories, but was essentially limited to the polishing steps. Grinding before polishing still was done by the conventional manual or mechanical methods. (See, for example the report by Nicholson and Williamson.[7]) Hopkins and Peterson[8] in their study of vibratory-polishing variables found that the method gives surfaces of excellent quality, but they also discussed some undesirable factors such as the large variation of metal removal with changes in vibrational amplitude. They expected an improvement from an increase in horizontal motion and decrease in vertical motion.

In Germany, Petzow, Gessner, and Hölscher[9] were able to grind specimens on a vibratory polishing apparatus. By carefully cementing abrasive paper to a glass plate and grinding either wet or dry, and by adjusting the amplitude of the vibration and the weight attached to the specimen, they succeeded in simultaneously grinding materials of different hardnesses. Their polishing method did not differ significantly from the American procedure.

This Laboratory is investigating a great variety of so-called exotic materials. They may consist of a hard and extremely brittle phase imbedded in a soft or refractory metal matrix, they may be diffusion couples wherein hard and soft diffusion bands neighbor each other and where internal boundaries must be clearly outlined, they may be high-purity metals containing trace amounts of residual second phases, they may be alpha-active materials that must be handled in gloveboxes, they may be materials wherein microcracks must be distinguished from other faults, or they may be single crystals. In all of these cases, vibratory metallographic-specimen preparation appears to be a highly promising method, provided one really controls all the important variables. This is achieved by: (1) doing away with the uncontrolled movement of the specimens in the apparatus, and providing a series of fixtures that guide the specimen in a predetermined manner;

(2) controlling the horizontal and vertical components of vibration, and changing the springs and the weight of the base of the apparatus to conform to the specific requirements of grinding or polishing; and (3) controlling the pressure on the sample, and providing a suitable, carefully-centered load.

The result is an automatic specimen-preparation process that, after machining* of the specimen and vibratory grinding of the backside of the mount to render the flat faces parallel, consists of:

1. Vibratory grinding of the specimen itself in a single operation,
2. Intermediate polishing,
3. Final polishing.

This report describes the fixtures that were devised for the three steps of vibratory grinding, intermediate polishing, and final polishing and gives the reasoning that led to each device. The method has been successfully applied to a variety of materials. Some significant results are shown at the end of this report. The method is now used on gamma-active materials in our hot cells and on alpha-active materials in our plutonium laboratory.

A Syntron Model LP-01 Type C Vibratory Polishing Apparatus was used in all our experiments.[10]

Vibratory operation of the commercial polishing apparatus is based on the tuned-spring system. An electromagnet, that provides the motive power, is interposed between the horizontal polishing bowl and the heavy base of the machine (see Fig. 21). The armature is affixed to the polishing bowl by means of an adapter plate and a mounting plate. The stator (of E-frame construction) is mounted to the base by adjustable bolts. Four gangs of four flat-springs each, position the bowl above the base and separate the armature and stator by a small air gap.

As the electromagnet is pulsed by a 60-cps, half-wave, rectified current, a vertical motion is imparted to the bowl-and-spring combination, and a forced vibration is impressed on the four gangs of flat springs. Since the springs are inclined 75° to the base, the combined effect is a helical motion of the polishing surface. The polishing bowl moves down and clockwise on the pulse and moves up and counterclockwise when the pulse dies. Mounted and properly weighted specimens are caused to travel around the periphery of the bowl. While the bowl is moving down and clockwise, the bowl drops away and the weighted mount falls by gravity to a new position on the bowl. However, when the bowl returns to its original position, it

*Machining is generally unnecessary except in glovebox work with plutonium materials.

exerts a force on the weighted mount. The magnitude of the force exerted depends on the mass of the weight fastened to the specimen mount. Since the weighted mount has an inertial mass, the mount will not move immediately as the bowl changes its direction of motion. This results in a short polishing stroke with the remaining portion of the pulse accounting for a small horizontal displacement of the specimen. The observable motion of the specimen results from this increment of displacement of a few thousands of an inch, which is repeated 3600 times a minute.

STEP I: VIBRATORY GRINDING

The initial exploratory work with slurries of 400- and 600-grit, silicon carbide abrasives with a variety of cloths proved ineffective for removing metal at a practical rate. Coarse, abrasive grit, because of its larger size, rolls under the weight of the specimen. Grinding action with such grit is slight. When a loose slurry was used, much of the abrasive was imbedded in the specimen surface. The grinding marks were non-uniform, and the metal was worked. After the use of slurries was abandoned, attention was turned to fixed abrasives, such as the abrasive paper discs commonly used for wet-grinding on rotating wheels. The selection of a paper disc with fixed abrasive resulted in a greater rate of metal removal. The grinding medium, however, aggravated two characteristic properties of the vibratory method:

1. Single-specimen mounts tend to overtake one another and to clatter when tracking at the periphery of the bowl.

2. A "bouncing" motion is imparted to the specimen by an interaction with the vibrating bowl.

The overtaking tendency by itself normally decreases only the rate of metal removal. Also, parallelism of the mount faces was not maintained, particularly when a relatively coarse abrasive was used. When a coarser abrasive medium was used, this tendency also resulted in embedding abrasive particles into the specimen. Abrasive particles were not embedded when a fine abrasive such as alumina was used. These effects were remedied by devising a lightweight fixture to confine and separate the mounts. The overtaking tendency was thereby prevented, and furthermore, the fixture assured positive flat contact of the specimen with the abrasive disc by holding the mount face parallel to the grinding surface.

The "bouncing" motion of the specimen was attributed to a "grabbing" tendency of the fixed abrasive and to an excessive vertical component of motion of the polishing bowl. By reducing the weight of the top of the unit as much as design would permit, and compensating for this weight reduction with a softer vibrational spring system, the vertical component of the vibratory motion was reduced. The reduction of vertical amplitude and the practical elimination of the attendant "bouncing" solved the basic problem of grinding of metallographic specimens.

Description of Equipment

The grinding fixture, shown in Fig. 1, is of all-Lucite construction and was designed to use an 8-in.-diam, abrasive paper disc. This fixture, shown schematically in Fig. 2, consists of a turret in which specimens and their weights are confined, and a bowl in which grinding is performed. The bowl serves as a support and a guide for the rotating turret. The rotating

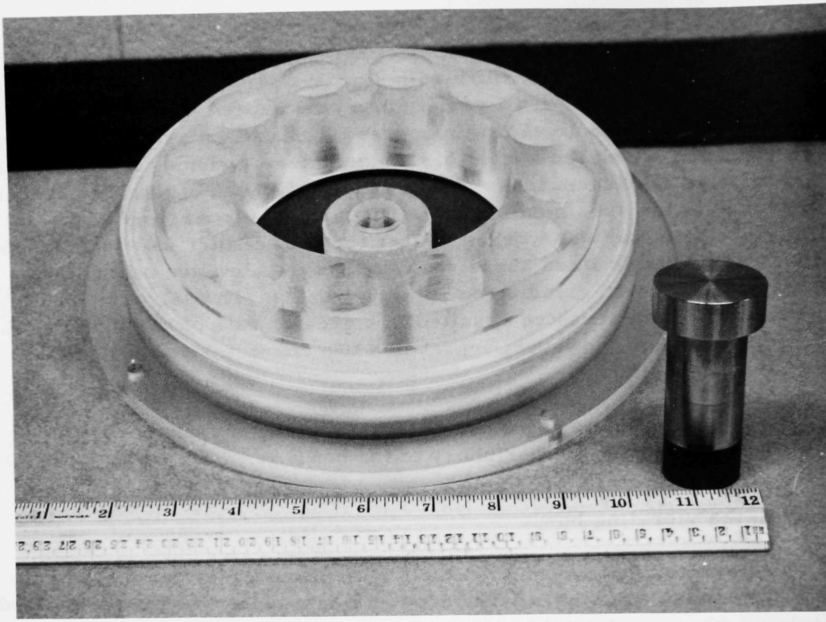

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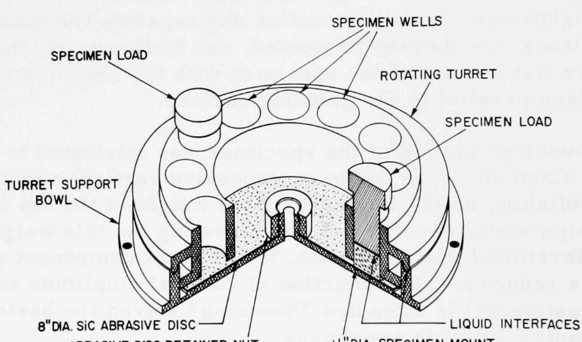
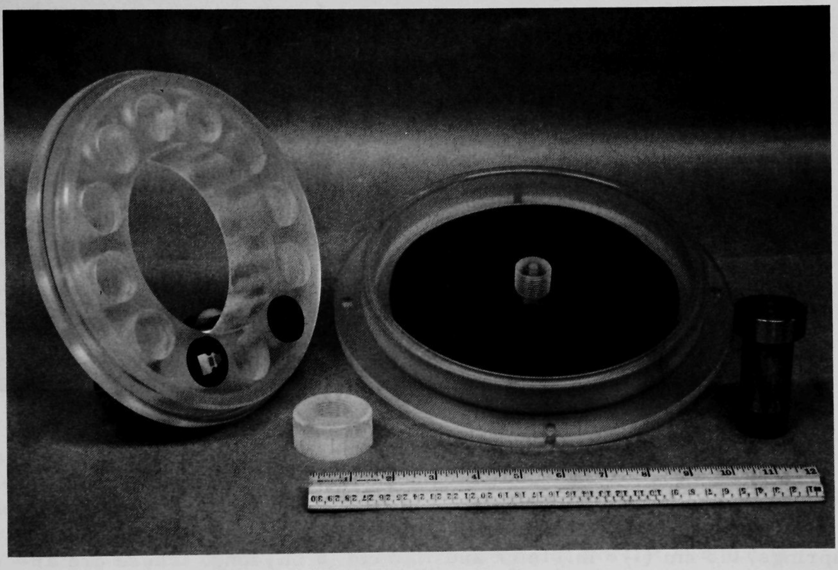
Fig. 1. Grinding Fixture and 500-g Weight



36100

Fig. 2. Schematic Cutaway of Vibratory Grinding Fixture

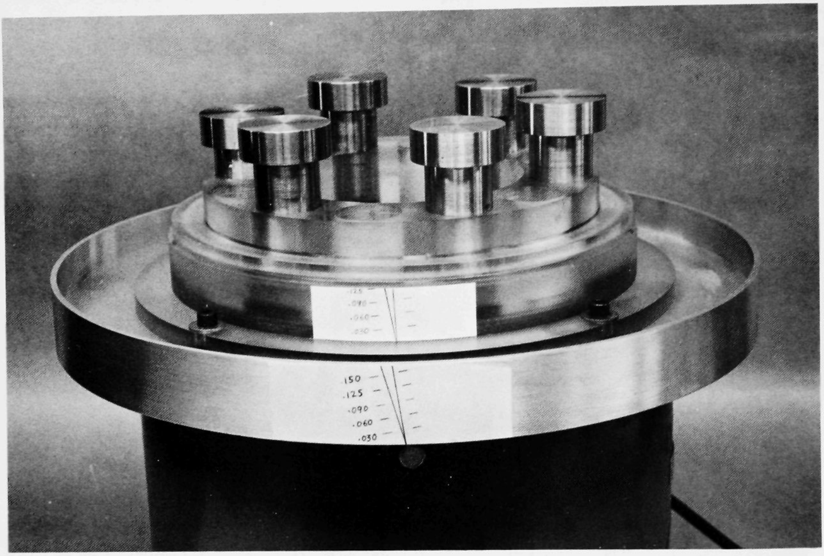
turret is essentially an annular ring with 12 bored holes in which 3.2-cm (1.25-in.)-diam, mounted specimens are constrained when they move over the abrasive disc. The turret is positioned over the abrasive disc (but not in contact with it) by a peripheral, low-friction track that is lubricated with ethylene glycol. (Ethylene glycol is used because of its nonevaporating property and its miscibility with water and with alcohol.) A silicon carbide abrasive disc with a 2.5-cm (1-in.)-diam, punched hole is placed in the center of the bowl, as shown in Fig. 3, and is secured by means of a Lucite retainer nut. (The Lucite nut has recently been replaced by a stainless-steel nut.) The abrasive disc is not cemented to the bowl. It is merely held down at its center. A recess is machined into the bowl at the periphery of the abrasive disc. This recess traps abraded material and any dislodged abrasive.



36183

Fig. 3. Components of Vibratory Grinding Fixture

The grinding fixture, with six of the specimen wells loaded, is shown in Fig. 4, mounted on a Syntron vibratory base. Weights, each weighing 500 g, are shown in position on top of the mounts. The fixture is fastened to an aluminum adapter plate, which also functions as a bowl to contain possible spatter. When one is grinding or polishing radioactive materials, this precaution is necessary. Smooth and effective grinding in this bowl and rotating-turret device necessitated the modification of the standard vibratory unit. A decrease in the vertical component of motion of the system without too great a loss in horizontal displacement was desired. The vertical component of motion was decreased by reducing the mass of the top fixture and by using softer springs. Weight was reduced by constructing the bowl and turret from Lucite and the bowl-plate from 6061-T6 aluminum alloy. The total weight of the grinding fixture and the adapter plate was reduced from the original 10.6 kg (23 lb 7 oz) of the commercial unit to 4.3 kg (9 lb 8 oz).



36187

Fig. 4. Partially Loaded Grinding Fixture Fastened to an Aluminum Bowl-plate

The commercial Syntron vibratory unit is provided with flat springs, 0.3 cm (1/8 in.) thick and 15.2 cm (6 in.) long. These are arranged in four gangs of four springs each and are inclined 75° from the base of the machine. A trial-and-error approach was adopted in determining the balanced, softer-spring system required. This was accomplished by varying the size and number of springs in each gang of springs. Two rules must be adhered to when modifying the spring system:

1. The spacers between springs must be placed symmetrically.
2. Opposite spring gangs must have the same spring arrangement.

The spring system, which was adopted for the grinding device weighing 4.3 kg, consists of four flat springs, 1.6 mm (0.063 in.) thick and

15.2 cm (6 in.) long, in each of the four gangs. Each four-spring gang is arranged symmetrically; the outer two spaces are fixed at 1.6 mm (0.063 in.), and the inner space at 3.2 mm (0.125 in.). A nominal air-gap setting of the electromagnet of 0.4 mm (0.016 in.) proved adequate. This balanced arrangement of softer spring system and lighter top resulted in a maximum vertical displacement of 0.64 mm (0.025 in.). Table I compares displacement amplitudes as a function of rheostat settings for the modified unit and the standard Syntron unit. Maximum vertical displacement occurred at a rheostat setting of 50 and a horizontal displacement of 1.65 mm (0.065 in.) for the modified unit. Increased rheostat settings increased the horizontal displacement with little increase in vertical displacement.

TABLE I. A Comparison of Displacement Amplitudes as a Function of Rheostat Settings

Rheostat Setting	Modified Unit: Horizontal Displacement (mm)	(in.)	Vertical Displacement (mm)	(in.)	Vertical Displacement / Horizontal Displacement	Standard Vibratory Unit: Horizontal Displacement (mm)	(in.)	Vertical Displacement (mm)	(in.)	Vertical Displacement / Horizontal Displacement
10	0.64	0.025	0.13	0.005	0.20	1.50	0.060	0.75	0.030	0.50
20	1.1	0.045	0.25	0.010	0.22	1.90	0.075	0.8	0.032	0.43
30	1.4	0.055	0.38	0.015	0.27	2.08	0.082	0.9	0.035	0.43
40	1.52	0.060	0.58	0.023	0.38	2.24	0.088	0.95	0.037	0.42
50	1.65	0.065	0.64	0.025	0.38	2.29	0.090	0.95	0.038	0.42
60	1.90	0.075	0.64	0.025	0.33	2.34	0.092	0.95	0.038	0.41
70	2.16	0.085	0.64	0.025	0.29	2.46	0.097	1.0	0.040	0.41
80	2.29	0.090	0.64	0.025	0.28	2.67	0.105	1.0	0.040	0.38
85	2.29	0.090	0.64	0.025	0.28	2.67	0.105	1.0	0.040	0.38

Procedure

The vibratory grinding step is standardized to 600-grit silicon carbide abrasive discs. A 200-cc mixture of 40% ethylene glycol with methanol, ethyl alcohol, or water is used as the lubricant. Sectioning of the specimen and initial mount preparation are important to the vibratory grinding step in that the surfaces of the mount must be flat and parallel to within 0.05 mm (0.002 in.). True parallelism of the flat surface is easily achieved by the vibratory grinding of the backside of the mount. This is done in the grinding fixture, as described here. The abrasive disc is discarded after this step. The mounted specimens are then dropped, face down, into the specimen wells of the rotating turret. Approximately 4 cc of ethylene glycol are placed on top of each mount. A 500-g weight is inserted into the well on top of the specimen displacing the ethylene glycol into the clearance between the mount and the well. The displaced glycol provides a cushion between the weight and the Lucite well and also provides a thin liquid film between the weight and the mount. The liquid film couples the mount and the weight and enables them to move vertically in unison. The film also facilitates removal of the specimen after the grinding operation; and since no clamping weights or screwed-on weights are used, the film makes for rapid sample removal and replacement. With this arrangement, the mount height is not important, although a 1.9-cm (0.75-in.)-high, Bakelite mount appears to be convenient.

A horizontal displacement amplitude of approximately 1.52 mm (0.060 in.) measured at the 24.1-cm (9.5-in.) outside diameter of the Lucite support bowl, results in turret rotation of about 5 to 7 rpm. While this displacement is not the maximum that is attainable on vibratory units, it does promote uniform and smooth grinding action in this adaptation. A grinding time of 1 hr is the maximum allowable. Grinding is practically complete after the first 30 min. Additional grinding time has a smoothening effect; but grinding longer than 1 hr can be detrimental because of abrasive paper wear and work-hardening of the specimen surface. Although grinding striations produced by this method are directional, the results are excellent, particularly with respect to the retention of inclusions. The presence of directional grinding striations aids readily in determining when the subsequent step is completed.

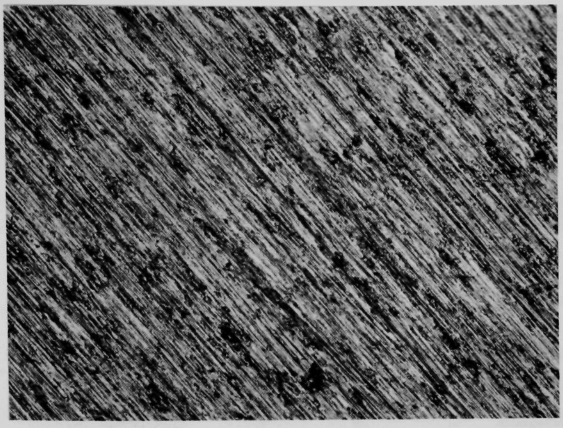
Results and Conclusions

Examples of some of the as-ground surfaces obtainable with the rotating turret grinding fixture are shown in Figs. 5, 6, and 7. All specimens were ground for 1 hr, each under a 500-g load.

Figure 5 at 100X compares the as-ground surfaces produced on 400-grit SiC papers (a) by hand-grinding on a rotating wet wheel and (b) by vibratory grinding. Note the smoothness and evenness of grinding striations and the absence of smeared metal in the vibratorily ground sample.

Figures 6 and 7 show the vibratory-ground surfaces produced by 600-grit abrasive discs. Figure 6(a) indicates that a comparable surface can be produced with 600-grit silicon carbide abrasive and that no real advantage is gained by first grinding on a 400-grit abrasive disc. Metal removal appears to be the same for both grades of abrasive. Figure 6(b) shows good edge-preservation. Cracks at and near the edge are clearly revealed in the as-ground surface. Figures 6(c) and 6(d) show the excellent retention in the as-ground condition of friable materials at the surface and the grain boundaries. Figure 7 (thorium and thorium-uranium alloys) is included to show the retention of inclusions and the absence of disturbed metal attained in specimens having a range of hardnesses. In particular, Fig. 7(d) shows the presence of phases not normally in evidence in this stage of sample preparation.

No attempt was made to determine the rate of metal removal to a high degree of accuracy. All measurements were done with a hand micrometer. Table II is a partial record of specimens that were vibratory-ground, and correlates metal removed with the hardness of the specimens.



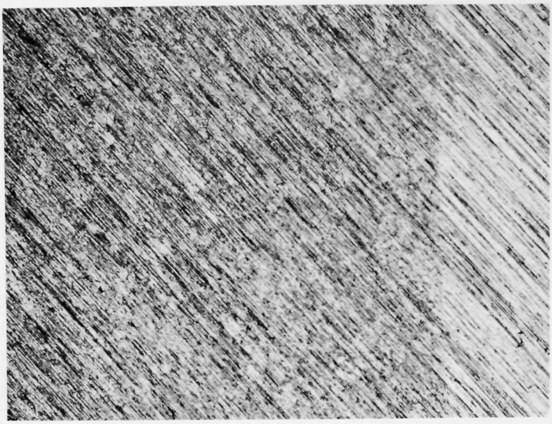
36194 100X

(a) Titanium hand-ground on a rotating wet wheel; 400-grit silicon carbide abrasive disc; shows the coarseness of the abraded surface and areas of smeared metal.

36196 100X

(b) Titanium vibratory-ground on a 400-grit silicon carbide abrasive disc, 1 hr under a 500-g load; shows the smoothness and evenness of striations and the absence of smeared metal.

Fig. 5. Comparison of Hand-grinding on a Rotating Wet Wheel and Vibratory Grinding



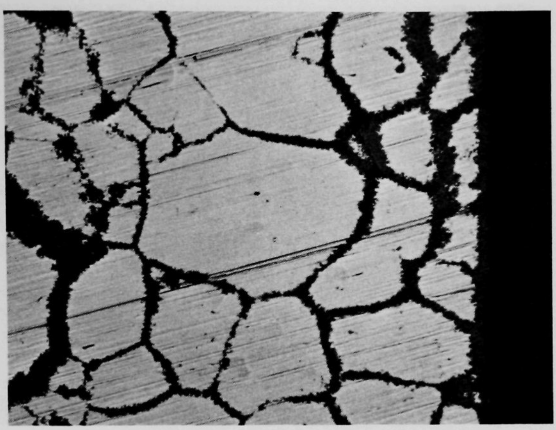
36198 100X

(a) Titanium dip-coated with aluminum, vibratorily ground on a 600-grit silicon carbide, abrasive paper disc. The surface produced indicates that no real advantage is gained by first grinding on a 400-grit silicon carbide paper. [Compare with Fig. 5(b)].

36200 250X

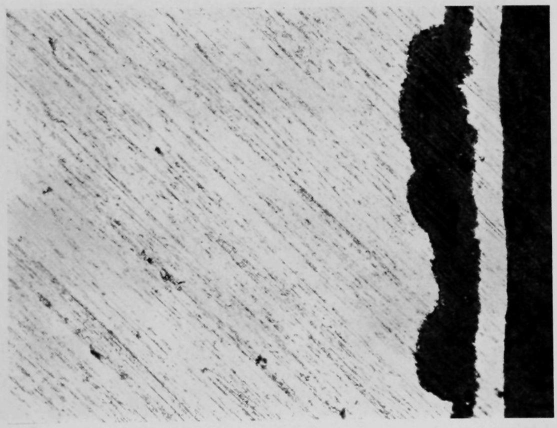
(b) Type 304 stainless steel shows good edge preservation; cracks at and near the surface are clearly revealed.

Fig. 6. Examples of Surfaces Produced by Vibratory Grinding on 600-grit Silicon Carbide Paper for 1 hr under 500-g Load



36478 250X

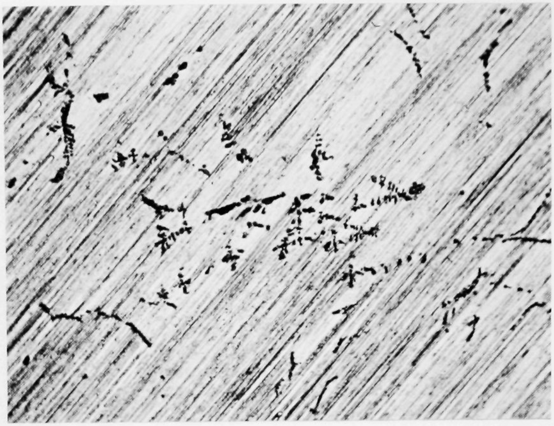
(c) "A" nickel, corrosion-tested, shows retention of friable material at the surface and at the grain boundaries.



36476 250X

(d) Aluminum alloy (X8001), corrosion-tested; the corrosion layer is protected by a strip of aluminum foil placed onto the surface before mounting.

Fig. 6. Examples of Surfaces Produced by Vibratory Grinding on 600-grit Silicon Carbide Paper for 1 hr under 500-g Load (Contd.)



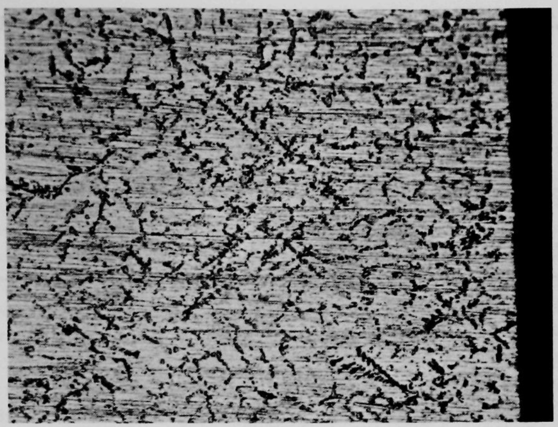
36201 250X

(a) Commercial-grade thorium with high-impurity content; shows good retention of hard inclusions in a soft matrix.

36202 250X

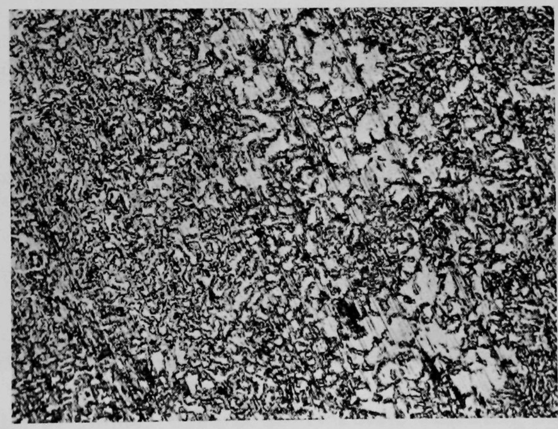
(b) High-purity thorium shows the clean as-ground surface obtained with a very soft material.

Fig. 7. Examples of Vibratorily Ground Thorium Samples. All specimens are as arc-melted. Grinding was done on 600-grit silicon carbide paper for 1 hr with 500-g load.



36203 250X

(c) Th-10w/oU



36204 250X

(d) Th-40w/oU alloy shows presence of phases not normally seen at this stage of sample preparation.

Fig. 7. Examples of Vibratorily Ground Thorium Samples. All specimens are as arc-melted. Grinding was done on 600-grit silicon carbide paper for 1 hr with 500-g load. (Contd.)

TABLE II. Metal Removal as a Function of Sample Hardness

Load: 500 g.
Lubricant: 200 cc of a methanol 40% ethylene glycol mixture.
Abrasive: 600-grit SiC paper disc (8-in. diam).
Time: 1 hr.

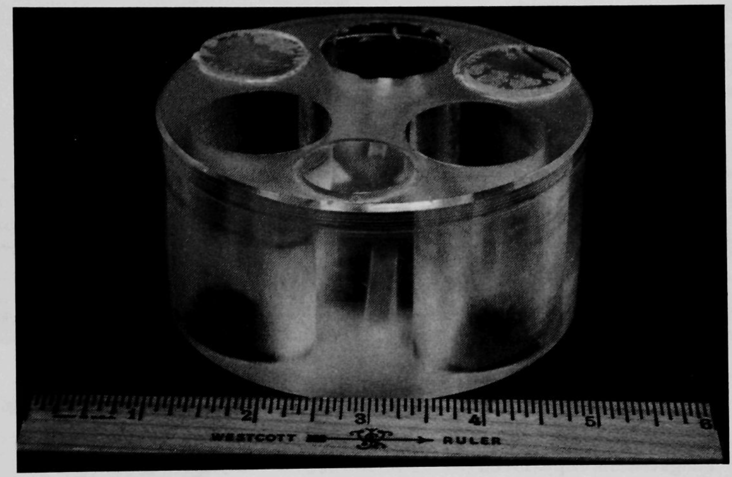
Specimen	Hardness	Metal Removed (mm)	Metal Removed (in.)
Commercial thorium	-	0.127	0.005
Crystal bar thorium	DPH 46	0.076	0.003
Th-2w/oU	-	0.076	0.003
Th-10w/oU	R_b 30	0.076	0.003
Th-40w/oU	R_b 64	0.051	0.002
Th-50w/oU	R_b 78	0.051	0.002
304 stainless steel	R_b 87	0.051	0.002
Tungsten	R_c 40	0.048	0.0015
Cobalt-Boron alloy	$>R_c$ 70	0.030	0.0012

A diamond-impregnated aluminum disc (3M Company, fine grade) was also tried. This resulted in a coarse-ground surface with insignificant metal removal. Vibratory grinding was also done with a Pellon disc (grade PAW) impregnated with 15-micron diamond abrasive. Lubricant in this trial was Hyprez oil. In this case, a grinding time of 1 hr resulted in a satisfactory surface with 2.5 to 6.3 microns (0.1 to 0.5 mil) removed from tungsten-UO_2 cermet specimens.

STEP II: INTERMEDIATE POLISHING

Description of Equipment

The commercial vibratory unit is used without modification in the second, intermediate, step of polishing. To facilitate its use and to allow for long-time or overnight polishing of specimens, a three-hole holder (shown in Fig. 8) is used. Three spacing lugs on the underside minimize contact between the holder and the polishing cloth. Single-specimen holders track only along the periphery of the bowl, resulting in a wearing out of the cloth at this location. Also, single specimens with clamped-on or screwed-on weights have the tendency to overtake each other with resultant clattering and a slowdown in polishing action. The three-hole holder eliminates the danger of specimens overturning and tearing the cloth. Since the holder rotates as it travels around the bowl, a large portion of the area of the polishing cloth is utilized and directional polishing is minimized.



38661

Fig. 8. Holder Used in Intermediate Polishing Step

Procedure and Results

After having been vibratorily ground and thoroughly cleaned to remove loose 600-grit contamination, the Bakelite-mounted specimens are placed into the wells of the three-hole holders used with the commercial vibratory unit. The vibratory unit can accommodate four such holders. This permits all 12 specimens to be transferred, after grinding, to the intermediate polishing step. In most cases, 500-g weights are used to load the specimens, but 1000-g weights have been used to polish cermets with a tungsten matrix.

The abrasive slurry consists of 15 to 20 g of Linde B alumina suspended in a mixture of 20 v/o ethylene glycol with either water or methanol. A quantity of 400 cc is maintained in the bowl. This mixture of a carrier liquid and ethylene glycol appears to be the optimum and has the advantage that when the carrier liquid evaporates, the ethylene glycol remains behind. The presence of the ethylene glycol permits intermittent operation of the vibratory units without caking of the polishing slurry. To make the unit operational again, it is only necessary to replenish with water or alcohol to the required level. This results in a saving of expensive polishing cloths. A nylon cloth underlayed with a less expensive Met-cloth is the lap material.

To determine the proper amplitude for polishing, the unit is allowed to operate initially with the rheostat set at its maximum. Then the rheostat is turned back slowly to decrease the amplitude. When the holders begin to rotate counterclockwise while orbiting at the periphery of the bowl, the amplitude is correct and polishing takes place. Frequently the weighed specimens rotate within the holder. Polishing time and displacement amplitude vary with the type of specimens being polished and the weights used. Some of the polishing times and weights used are summarized in Table III.

TABLE III. Summary of Final-polished Specimens Listing Time and Weight for Each Step

Specimen		Vibratory Ground		Intermediate Polish		Final Polish	
Fig. No.	Identification	Time (hr)*	Load (g)	Time (hr)	Load (g)	Time (hr)	Load (g)
14	Commercial thorium	1	500	4	500	2	0
15	Th-50w/oU	1	500	3	1000	1	250
16	304 stainless steel, sensitized	1	500	2	500	2	125
17	Tungsten on copper rod	1	500	6	1000	2	0
18	UO_2 in tungsten	1	500	16	1000	2	0
19	Co-5.5w/oB	4	500	Over-night	500	3	0

*Changing the abrasive disc every 1/2 hr, while time-consuming, minimizes work-hardening effects.

Generally, the use of the unmodified unit in the intermediate step should be restricted to materials with polishing characteristics similar to steel or with hardness values equal to, or greater than, those of steel. Because of their resistance to abrasion, very soft materials proved exceptionally difficult to polish by this method. Intermediate polishing with an abrasive of larger average-particle size (0.3 micron--Linde A) was attempted. Metal removal from specimens of pure thorium was insignificant. The rate of metal removal was determined by measuring the diagonals of DPH impressions before and after polishing. The amount of metal removed was calculated from the known geometry of the indenter. Four microns/hr were removed under a 500-g load on a nylon cloth. Polishing on microcloth under the same conditions of weight and abrasive removed only 3 microns/hr. The surfaces produced were not satisfactory. In contrast, more than 7 microns of material were removed from a specimen of a 50 w/o thorium-uranium alloy by polishing for 1 hr on nylon cloth. The exact amount removed is not known since the 7-micron depth of the hardness indentation was exceeded. The rates of metal removal of the other materials polished in the intermediate step were not determined. Figure 9 shows the scratch size and distribution developed in a specimen of pure thorium under a 250-g load. Similar results were obtained using a 500-g or 1000-g load. These observations suggest that specimens that are allowed to track freely at the periphery of the bowl do not make positive contact with the polishing cloth and momentarily tend to ride the edge of the mount. Effective cutting action would then be decreased. Perhaps this type of action is inherent in vibratory polishing machines. The effect of this action is more noticeable in the soft materials. Harder specimens do not seem to be seriously affected, and the slight polishing scratches or directional striations resulting can readily be removed in the final-polishing device.

36785 250X

Fig. 9. Pure Thorium; Scratch Size and Distribution Resulting from Intermediate Step of Polishing a Very Soft Material

STEP III: FINAL POLISHING

Description of Equipment

The final-polishing fixture (shown in Fig. 10), also constructed of Lucite, is standardized for 30-cm (12-in.)-diam, polishing cloths. This fixture is an adaptation of the three-hole holder used in the intermediate polishing step and features a rotating carrier with a center-mounted frictioning device. Figure 11 is a schematic cutaway section of the rotating carrier and friction loading device and shows the placement of specimens within three-hole holders. The three-hole holder fits closely into the

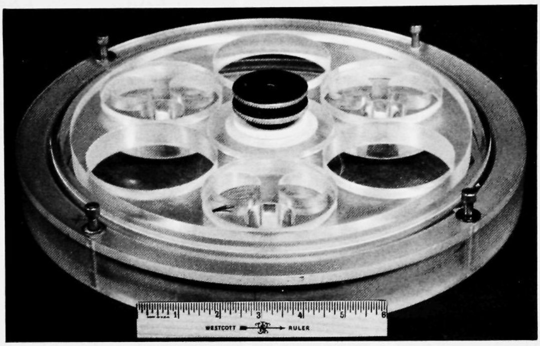
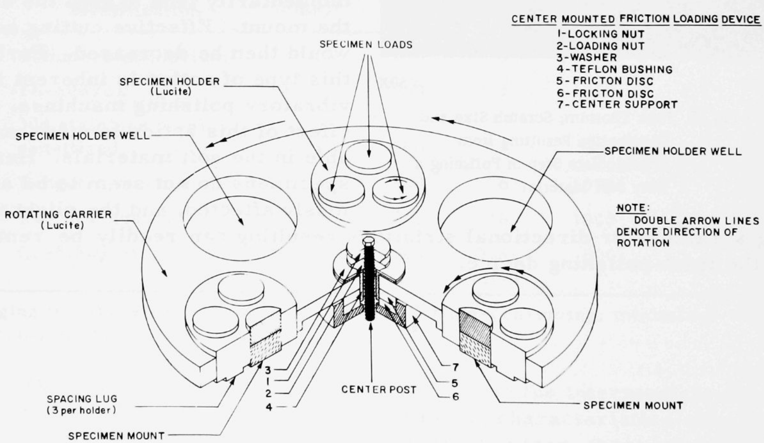


Fig. 10

Final-polishing Fixture and Bowl

38658



36716

Fig. 11. Schematic Cutaway of Vibratory Final-polishing Fixture

rotating carrier but rotates independently. A commercial vibratory unit[1] is used, but the stainless-steel bowl and retaining ring were replaced with the final-polishing fixture, a Lucite bowl, and an adapter plate of the same total weight. Successful polishing with this fixture depends on the restraining effect of the center-mounted snubbing device, which, when properly loaded by tightening the loading nut and locking it, slows the carrier down, and thereby introduces counterclockwise rotation of the specimen holders. Figure 12 is a disassembled view and shows the rotating carrier and a clamping ring positioned over the bowl. The ring holds a cloth by means of an O ring and groove, and stretches and clamps the cloth securely into a recess at the periphery of the bowl.

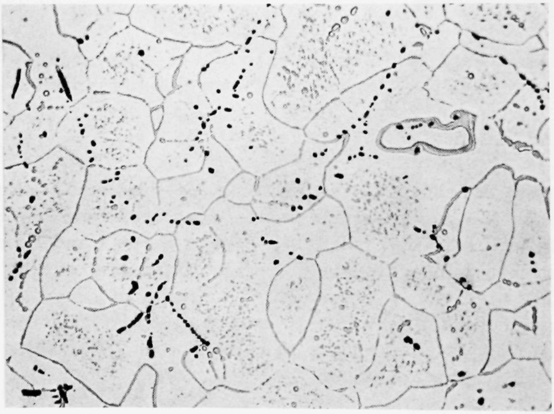
38659

Fig. 12. Components of Vibratory Final-polishing Fixture (Unassembled)

<u>Procedure and Results</u>

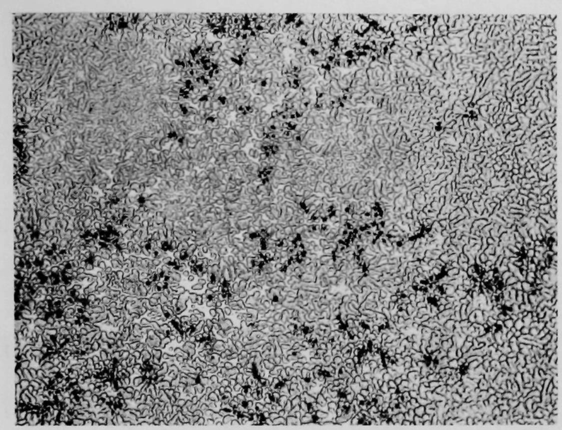
Since the bored specimen wells (1.250-in. diam) have the same size as those used in the two prior steps, the specimens may be quickly and easily transferred for final polishing. As in Steps I and II, ethylene glycol is used as the liquid interface between specimen and weight. The abrasive slurry consists of 15 to 20 g of Linde B alumina in 200 cc of a mixture of 20 v/o ethylene glycol with water or methanol. Microcloth, because of its short and stiff nap, is used as the polishing cloth. Polishing can be done without any load on the specimen.

When specimens are polished without a load, one of the holders must be loaded with a dummy weight of at least 250 g in each hole to promote rotation of the carrier. In this application, the bowl is operated at maximum amplitude. If a new cloth has been installed, the carrier is allowed to operate at maximum rotational speed for a short time to aid in conditioning the cloth. For proper and nondirectional polishing, the snubbing nut is gradually tightened until the three-hole holders begin to rotate. The specimens will tend either to oscillate or to rotate counterclockwise within their holes. Polishing time for most specimens will vary between 1 and 2 hr. The use of excessive weights will result in relief polishing. Figures 13 through 18 are photomicrographs of a variety of materials successfully polished to a striation- and scratch-free surface. Table III summarizes the steps required to prepare these materials metallographically by the three-step vibratory method. Pure thorium specimens (whether arc-melted or as-received, crystal-bar stock) proved difficult to final-polish. As noted earlier in Fig. 9, this may be due to improper surface preparation in the intermediate step. Figure 13 is a specimen of commercial-grade thorium. The presence of a high-impurity phase probably contributed to successful vibratory preparation of this material. The harder thorium alloys, examples of which are shown in Fig. 14, did not pose any particular problem. Highly polished surfaces were easily attained. Figure 15 shows a sensitized, Type 304 stainless-steel specimen at a magnification of 500X. Figure 16, at a magnification of 200X, shows tungsten electroformed onto a copper rod, and is shown as an example of polish attained on materials of dissimilar hardness and properties. The copper was etched with a 10% ammonium persulfate solution. The black area between the copper and tungsten is not a step due to relief polishing, but is a void or separation existing between the two materials. Cermets of tungsten and UO_2 are routinely prepared by this method with the intermediate step done overnight. Figure 17 shows UO_2 particles in a sintered tungsten matrix. The



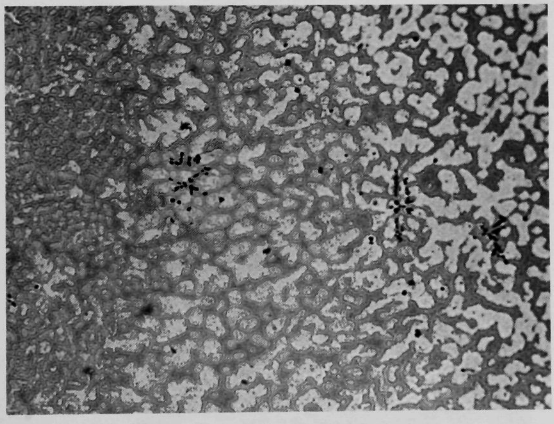
36480 250X

Fig. 13. Commercial Thorium with High-impurity Content; As-polished Surface; Polishing Time: 2 hr; Unweighted



39054 250X

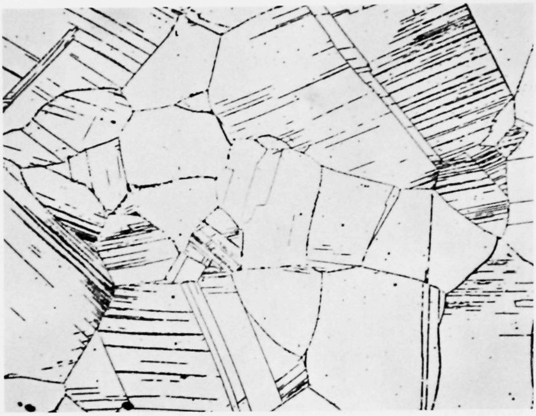
(a) As-polished surface



39057 250X

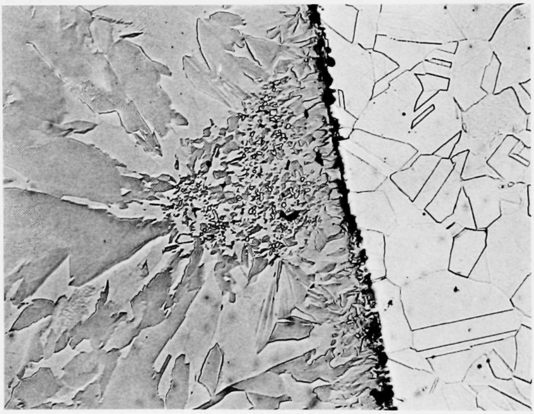
(b) As-polished surface with a light air-etch

Fig. 14. Two Examples of an Arc-melted Th-50w/oU Alloy, Polished 1 hr under a 250-g Load. Photomicrographs were taken of different areas of the same specimen.



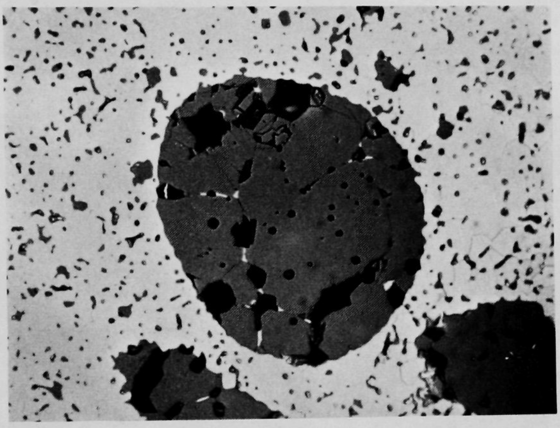
39004 500X

Fig. 15. Type 304 Stainless Steel, Sensitized, Polished 2 hr under a 125-g Load, Etched Electrolytically in 10% Oxalic Acid



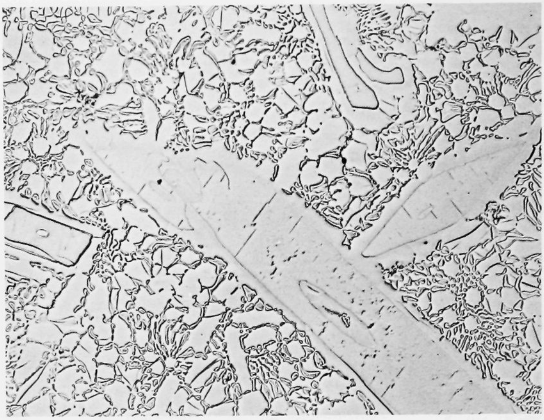
38874 200X

Fig. 16. Electroformed Tungsten on a Copper Rod, Polished 2 hr, Unweighted, Etched in 10% Ammonium Persulfate



38716 500X

Fig. 17. UO_2 Particle in a Sintered Tungsten Matrix. As-polished, unweighted, polishing time: 2 hr.



EI-1231 100X

(a) As-polished surface

EI-1232 100X

(b) As-polished, polarized-light photograph

Fig. 18. Co-5.5 w/o Boron, Melted at 1250°C, Annealed for 18 hr at 1000°C. Overall hardness, 1250-1375 DPH, RC > 70. Final-polishing Time: 3 hr. Unweighted.

cobalt-5.5 w/o boron alloy, shown in Fig. 18, is an interesting specimen because of its high hardness. The surface shown is as-polished for 3 hr, unweighted. Because of its high hardness, prolonged polishing did not produce a visibly disturbed surface. This is confirmed in the polarized-light photograph of the same as-polished surface. Vibratory grinding of this specimen took 4 hr under a 500-g weight and required four changes of abrasive paper.

Figure 19 shows the vibratory metallography installation in use at ANL. The vibratory grinder is on the left; intermediate polishing is done in the middle unit; final polishing is accomplished in the unit on the right. The three-step procedure used is outlined in the summary presented in the following section.

38657

Fig. 19. Vibratory Metallography Installation in Use at ANL

SUMMARY OF VIBRATORY GRINDING AND POLISHING PROCEDURES

Step 1: Vibratory Grinding

Abrasive:	400- or 600-grit silicon carbide paper.
Lubricant:	200 cc of a mixture of 60 v/o methanol or ethyl alcohol (low in water content), 40 v/o ethylene glycol.
Track Lubricant:	Ethylene glycol.
Load:	500 g, 1000 g for very hard specimens. After the specimen is placed into the hole of the guide ring, 4 cc of ethylene glycol are put into the hole with an eye-dropper. Then the weight is inserted. It displaces the ethylene glycol, which forms a lubricating film around the specimen and the weight and also between the specimen and the weight. The film between the specimen and the weight makes the two adhere to each other and facilitates their removal.
Time:	Maximum 1 hr. Most grinding completed in 1/2 hr.
Precautions:	To prevent trapping of hard particles, do not remove specimens for inspection during grinding. Use new silicon carbide paper each time.
Preparation:	Grind mount first on back side to obtain parallel end faces. Discard silicon carbide paper disc.

Step 2: Intermediate Polishing

Abrasive:	Linde B alumina on nylon cloth (napless) cushioned by Metcloth.
Lubricant:	80 v/o methanol or ethyl alcohol, 20 v/o ethylene glycol (400 cc).
Load:	500 g applied with 4 cc ethylene glycol as above.
Time:	3 to 6 hr, or overnight for hard specimens.
Precaution:	Maintain liquid level by adding alcohol to replace loss by evaporation. The presence of ethylene glycol permits intermittent use of polisher by preventing caking of the abrasive slurry. Different from grinding, specimen may be removed for inspection and replaced.

Step 3: Final Polishing

Abrasive:	Linde B alumina on microcloth (short nap).
Lubricant:	80 v/o methanol or ethyl alcohol, 20 v/o ethylene glycol (200 cc).
Load:	None or up to 250 g. Dummy weights of 250 g in another three-hole holder to promote rotation of carrier.
Time:	One to 2 hr.
Precaution:	Same as with intermediate polishing.

DISCUSSION

The new mechanical fixtures incorporated into the vibratory apparatus make the preparation of the metallographic specimen easier and produce surfaces of excellent quality with materials that heretofore were extremely difficult to handle. These improvements make it possible to introduce a large degree of automation into remote-handling and glovebox work. The fixtures developed may not represent the most economic design, but they establish the principle that controlled guidance of the specimens over the vibratory grinding and polishing surfaces are mandatory in the preparation of good specimen surfaces. Under such conditions, only a limited amount of work-hardening of the surface of the metal takes place, as evidenced by the appearance of inclusions and some structural features already visible after the grinding operation. Vibratorily polished specimens frequently require less severe etching to develop the structure than do manually prepared specimens, and the vibratory polishing minimizes the need for repeated polishing and etching.

The materials used in the construction were selected because of their light weight and not necessarily because of durability.

Much work needs to be done with regard to the selection of polishing cloths, abrasives, and carrier liquids for the abrasive. Ethylene glycol as a nonevaporating vehicle for the abrasive was given preference over other solvents because of the utilization of the vibratory method in gloveboxes with a dry inert atmosphere. Because of its lubricity, the presence ethylene glycol may have compromised the cutting action of the abrasive.

In general, the vibratory grinding approach proved successful on a wide variety of materials and also on the few composites that were available. A disadvantage of the method lies in the recurring track that the specimen takes over the abrasive disc. A hard material will actually groove the disc slightly. The greater wear imparted to the abrasive disc in a localized track leaves a visible raised section on the surface of softer materials. This does not allow the simultaneous grinding of materials having a great dissimilarity in hardness for long periods of time when the harder material is smaller in size than the companion specimens. In some instances, this effect can be removed in the intermediate polishing step.

The use of the grinding fixture in an intermediate step of polishing with an intermediate grade of abrasive should be investigated. Although the surface produced would then be characterized by fine directional striations, this would no be objectionable at this stage of specimen preparation. It has been observed that soft specimens coming off the intermediate polishing step with a surface of fine, directional, polishing striations (due to nonrotation of the holder) possessed a much cleaner-looking surface than soft specimens that had rotated. In such a fixture, the lap material is fastened around a center post and is allowed to expand radially outward. Since the lap material is not restrained at its peripher, it would have to be stiff enough to resist wrinkling under the gripping action of the weighted mounts. Also, it would have to be flexible enough to dampen out resonant vibrations. The abrasive used could either be fixed into the lap material, or else it could be a loose slurry, since it would be of relatively fine size.

Because metal removal occurs in minute increments, the preservation of inclusions and phases is good. Any method that confines the mounts and prevents their rotation, as does the grinding fixture, insures positive flat contact with the lap material. Uniform cutting action is thereby increased.

Although the intermediate and final polishing steps were carried out on unmodified, commercial, vibratory units, a further improvement in polishing action might have been achieved by reducing the vertical component of displacement. This can be done, as indicated earlier, by reducing the weight of the top fixtures and by compensating for the weight difference with a softer spring system. The spring system is readily accessible, as shown in Fig. 20, and a variety of symmetrical configurations can be arranged with any combination of the following changes:

1. Using fewer springs.
2. Using thinner springs.
3. Varying the spacing between springs.

In addition, a departure could be made from the center-mounted rotating carrier in the final-polishing fixture to a similar device that would be supported at its periphery as in the grinding device. Snubbing to restrain rotation and to introduce polishing action could then be done at the peripheral track. This type of arrangement would leave the center of the bowl free for a cloth holding-and-stretching device.

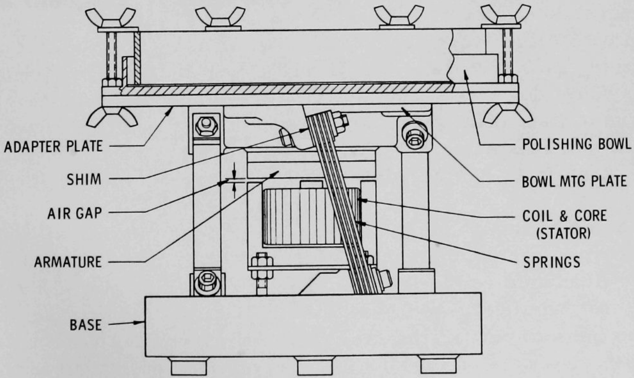


Fig. 20. Spring and Bowl Arrangement of Syntron Vibratory Polishing Apparatus

CONCLUSIONS

The application of the principle of controlled guidance of the specimens in vibratory polishing has brought significant advantages to the preparation of metallographic specimens by this method in addition to the advantages already noted by earlier investigators. The method is applicable to remote handling and glovebox work and produces specimens of high quality from a great variety of materials which otherwise are difficult to prepare.

ACKNOWLEDGMENTS

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